0.49 (2), 0.56 (2), 0.38 (2) and 0.38 (2), respectively, and were treated anisotropically, except for O(9″). The H-atom positions in the CH, CH_2 and CH_3 groups were generated, while those linked to O atoms were located in a difference Fourier map. Their positions were refined and included in the structure-factor calculations with a common isotropic temperature factor, U = 0.060 Å^2. Only one H atom for each water molecule was located. The high R value is due to the presence of disorder.

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71751 (24 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CD1053]

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D-*glycero*-D-*gulo*-Heptono- (I) and 2,7-Ditosyl-D-*glycero*-D-*gulo*-heptono-1,4-lactone (II)

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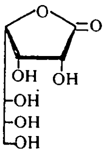
Abstract

The geometries of the lactone rings in the two structures are similar. Differences between $C_7H_{12}O_7$ (I) and $C_{21}H_{24}O_{11}S_2$ (II) occur in the conformation of the side chain with respect to the lactone ring, and in the crystal packing, with that of (I) being more influenced by hydrogen bonding. A weak intramolecular hydrogen bond of 2.718 (6) Å is present in (II).

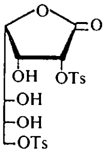
Comment

An investigation of the selective di-*O*-tosylation of aldonolactones and the selective di-*O*-mesylation of hexonolactones shows that the selectivity is good when the hydroxy groups at C(2) and C(3) are *cis* oriented. Furthermore, the selectivity is highest for the lactones which also have the side chain *cis* to the two hydroxy groups mentioned (Lundt & Madsen, 1992).

As tosyl and mesyl are good leaving groups the di-*O*-tosylates and di-*O*-mesylates can be used as substrates for nucleophilic substitution reactions. The ditosylation of D-*glycero*-D-*gulo*-heptono-1,4-lactone (I) gave the 2,7-di-*O*-tosylate (II) in good yield (64%). This yield is rather high considering that five O atoms are available for tosylation and could be a result of steric hindrance or intramolecular hydrogen bonding between some of the hydroxy groups. In order to see if a connection between conformation and yield could be found, the present structure investigations were carried out.

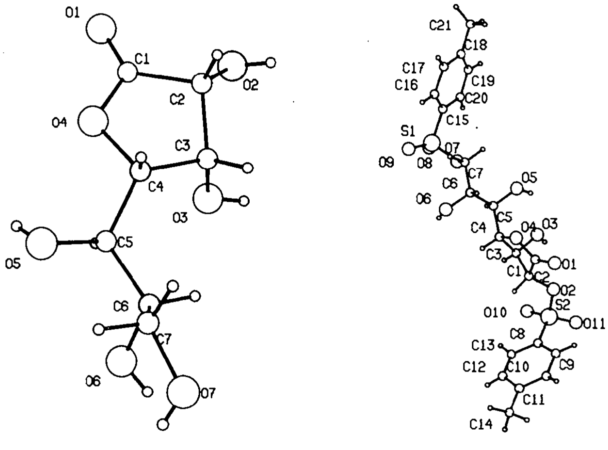


(I)



(II)

The commercial compound (I) (Sigma) was recrystallized from ethanol at room temperature. Compound (II) was prepared by literature methods (Lundt & Madsen, 1992). The reflecting power of the crystals of (II) was rather poor. The bond lengths and angles listed in Table 2 agree well with those observed in related structures. The labelling of the atoms is shown in Fig. 1.



(I) (II)

Fig. 1. View of the molecules with atomic labelling.

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The lactone rings are nonplanar. The phase of pseudorotation, P, and the amplitude of pucker, τ_m (Rao, Westof & Sundaralingam, 1981), for (I) and (II) are $P(\mathrm{I}) = 197.7\,(3)$, $P(\mathrm{II}) = 187.2\,(5)^\circ$ and $\tau_m(\mathrm{I}) = 31.9\,(1)$, $\tau_m(\mathrm{II}) = 40.0\,(3)^\circ$. Thus, the conformation of (I) is ${}_3E$ (the ideal value of P is 198° for ${}_3E$), while (II) exhibits a conformation between ${}^2_3T$ and ${}_3E$ (the ideal value of P is 180° for ${}^2_3T$). The corresponding puckering phase angles, φ_2, and the amplitudes, q_2 (Cremer & Pople, 1975), are $\varphi_2(\mathrm{I}) = 107.2\,(5)$, $\varphi_2(\mathrm{II}) = 97.6\,(9)^\circ$ and $q_2(\mathrm{I}) = 0.314\,(3)$, $q_2(\mathrm{II}) = 0.392\,(7)$ (the values of φ_2 for E and T are 108 and 90°, respectively). Similar ring conformations are found in other 1,4-lactones (Berman, Rosenstein & Southwick, 1971; Gress & Jeffrey, 1976; Kinoshita, Ruble & Jeffrey, 1981; Baird *et al.*, 1987). Differences between (I) and (II) arise in the conformation of the side chain with respect to the lactone ring. Selected torsion angles are listed in Table 2. In (I) the hydroxy group O(5)—H is oriented in a direction away from O(3), whereas in (II) the O(5)—H group is turned towards the O(3)—H group, giving rise to a weak intramolecular hydrogen bond, which is described in Table 3.

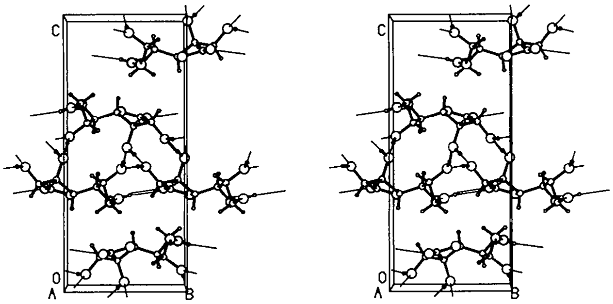

Fig. 2. Stereoscopic view of D-*glycero*-D-*gulo*-heptono-1,4-lactone (I); hydrogen bonds are drawn as thin lines.

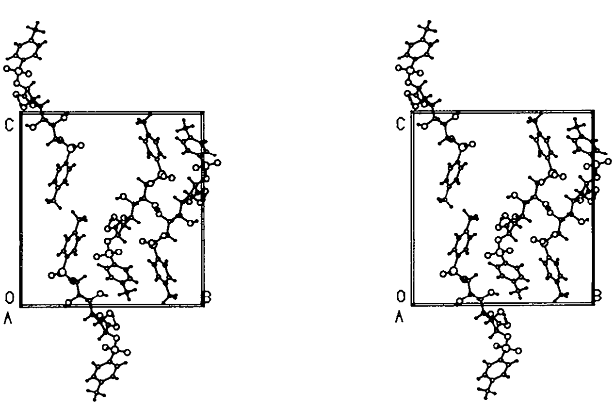

Fig. 3. Stereoscopic view of 2,7-ditosyl-D-*glycero*-D-*gulo*-heptono-1,4-lactone (II); hydrogen bonds are drawn as thin lines.

The crystal packing of (I) is influenced by hydrogen bonds, which are described in Table 3 and shown in Fig. 2. Each of the three hydroxy groups O(2)—H, O(5)—H and O(7)—H are bonded to a molecule related by translational symmetry in the **a** or **b** direction. In the packing of (II) shown in Fig. 3, the only hydrogen bond is O(3)—H···O(1), which bonds the hydroxy group to a molecule related by translational symmetry in the **a** direction.

Experimental

Compound (I)

Crystal data

$C_7H_{12}O_7$
$M_r = 208.17$
Orthorhombic
$P2_12_12_1$
$a = 6.612\,(1)$ Å
$b = 7.685\,(1)$ Å
$c = 17.191\,(2)$ Å
$V = 873.5\,(2)$ Å^3
$Z = 4$
$D_x = 1.583$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10\text{–}19^\circ$
$\mu = 0.14$ mm^{-1}
$T = 296$ K
Plate
0.45 × 0.14 × 0.04 mm
Colourless

Data collection

Enraf-Nonius CAD-4 diffractometer
ω scans
Absorption correction: none
4440 measured reflections
2208 independent reflections
1107 observed reflections [$I \geq 3.0\sigma(I)$]

$R_{\mathrm{int}} = 0.026$
$\theta_{\mathrm{max}} = 35^\circ$
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 12$
$l = -27 \rightarrow 27$
2 standard reflections
frequency: 240 min
intensity variation: none

Refinement

Refinement on F
$R = 0.039$
$wR = 0.040$
$S = 1.27$
1107 reflections
175 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F) + 0.0004|F|^2]$
$(\Delta/\sigma)_{\mathrm{max}} = 0.041$
$\Delta\rho_{\mathrm{max}} = 0.29$ e Å^{-3}
$\Delta\rho_{\mathrm{min}} = -0.24$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Compound (II)

Crystal data

$C_{21}H_{24}O_{11}S_2$
$M_r = 516.53$
Orthorhombic
$P2_12_12_1$
$a = 5.781\,(2)$ Å
$b = 19.526\,(3)$ Å
$c = 20.538\,(4)$ Å
$V = 2318\,(1)$ Å^3
$Z = 4$
$D_x = 1.480$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10\text{–}16^\circ$
$\mu = 0.28$ mm^{-1}
$T = 296$ K
Needle
0.08 × 0.05 × 0.05 mm
Colourless

Data collection

Enraf-Nonius CAD-4 diffractometer
ω scans
Absorption correction: none
7544 measured reflections
6725 independent reflections
2872 observed reflections [$I \geq 1.5\sigma(I)$]

$R_{\rm int} = 0.026$
$\theta_{\rm max} = 30°$
$h = 0 \rightarrow 8$
$k = 0 \rightarrow 27$
$l = -28 \rightarrow 28$
2 standard reflections frequency: 240 min intensity variation: none

Refinement

Refinement on F
$R = 0.071$
$wR = 0.046$
$S = 1.37$
2872 reflections
387 parameters

$w = 1/[\sigma^2(F) + 0.0002|F|^2]$
$(\Delta/\sigma)_{\rm max} = 0.22$
$\Delta\rho_{\rm max} = 1.09$ e Å^{-3}
$\Delta\rho_{\rm min} = 0.64$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for compounds* (I) *and* (II)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
(I)				
O(1)	−0.3220 (3)	0.6821 (3)	0.6330 (1)	0.0388 (6)
O(2)	0.0055 (3)	0.8316 (3)	0.5519 (1)	0.0344 (7)
O(3)	0.1791 (3)	0.5127 (3)	0.5194 (1)	0.0284 (6)
O(4)	−0.1122 (3)	0.4609 (2)	0.6559 (1)	0.0277 (6)
O(5)	0.0405 (3)	0.1286 (3)	0.6818 (1)	0.0313 (6)
O(6)	0.3282 (3)	0.0320 (3)	0.5596 (1)	0.0409 (7)
O(7)	0.6468 (3)	0.0537 (3)	0.6641 (2)	0.0427 (8)
C(1)	−0.1528 (4)	0.6249 (3)	0.6348 (1)	0.0256 (7)
C(2)	0.0408 (4)	0.7181 (3)	0.6143 (2)	0.0252 (8)
C(3)	0.1896 (4)	0.5693 (3)	0.5983 (2)	0.0232 (7)
C(4)	0.1077 (4)	0.4277 (3)	0.6513 (2)	0.0225 (7)
C(5)	0.1269 (4)	0.2397 (3)	0.6244 (2)	0.0232 (7)
C(6)	0.3435 (4)	0.1807 (3)	0.6088 (2)	0.0236 (7)
C(7)	0.4565 (5)	0.1309 (5)	0.6819 (2)	0.0384 (10)
(II)				
S(1)	0.5854 (3)	0.22593 (8)	0.17800 (7)	0.0483 (6)
S(2)	0.6173 (3)	0.51570 (8)	−0.22602 (7)	0.0503 (6)
O(1)	1.1955 (7)	0.5244 (2)	−0.0976 (2)	0.057 (2)
O(2)	0.7662 (7)	0.5255 (2)	−0.1609 (2)	0.048 (1)
O(3)	0.6009 (8)	0.4877 (2)	−0.0369 (2)	0.052 (2)
O(4)	1.0703 (7)	0.4330 (2)	−0.0424 (2)	0.045 (1)
O(5)	0.8146 (9)	0.4361 (2)	0.0703 (2)	0.053 (2)
O(6)	0.9517 (12)	0.2629 (2)	0.0226 (3)	0.058 (2)
O(7)	0.6471 (7)	0.2885 (2)	0.1347 (2)	0.049 (2)
O(8)	0.3426 (7)	0.2172 (2)	0.1697 (2)	0.067 (2)
O(9)	0.7320 (8)	0.1696 (2)	0.1627 (2)	0.065 (2)
O(10)	0.4525 (8)	0.4629 (2)	−0.2156 (2)	0.062 (2)
O(11)	0.5479 (8)	0.5840 (2)	−0.2409 (2)	0.062 (2)
C(1)	1.0576 (12)	0.4792 (3)	−0.0904 (3)	0.043 (2)
C(2)	0.8409 (12)	0.4652 (3)	−0.1297 (3)	0.045 (2)
C(3)	0.6837 (11)	0.4351 (3)	−0.0781 (3)	0.046 (2)
C(4)	0.8562 (12)	0.3930 (3)	−0.0403 (3)	0.044 (2)
C(5)	0.7990 (11)	0.3785 (3)	0.0308 (3)	0.039 (2)
C(6)	0.9507 (12)	0.3233 (3)	0.0605 (3)	0.041 (2)
C(7)	0.8931 (12)	0.3094 (3)	0.1306 (3)	0.042 (2)
C(8)	0.8209 (11)	0.4869 (3)	−0.2830 (2)	0.043 (2)
C(9)	0.9968 (13)	0.5285 (4)	−0.3030 (3)	0.058 (3)
C(10)	1.1471 (15)	0.5046 (4)	−0.3506 (4)	0.066 (3)
C(11)	1.1297 (13)	0.4408 (4)	−0.3782 (3)	0.055 (3)
C(12)	0.9477 (19)	0.4004 (4)	−0.3553 (4)	0.082 (4)
C(13)	0.8000 (15)	0.4225 (4)	−0.3091 (3)	0.070 (3)
C(14)	1.2893 (15)	0.4159 (4)	−0.4292 (3)	0.077 (3)
C(15)	0.6411 (11)	0.2536 (3)	0.2577 (2)	0.038 (2)
C(16)	0.8453 (13)	0.2351 (3)	0.2882 (3)	0.055 (3)
C(17)	0.8836 (15)	0.2580 (4)	0.3502 (3)	0.065 (3)
C(18)	0.7334 (18)	0.2991 (4)	0.3826 (3)	0.073 (3)
C(19)	0.5324 (16)	0.3163 (4)	0.3511 (4)	0.067 (3)
C(20)	0.4827 (13)	0.2950 (3)	0.2882 (3)	0.052 (3)
C(21)	0.783 (2)	0.3234 (4)	0.4510 (3)	0.108 (4)

Table 2. *Selected geometric parameters* (Å, °) *for compounds* (I) *and* (II)

	(I)	(II)
S(1)—O(7)	-	1.553 (4)
S(1)—O(8)	-	1.424 (4)
S(1)—O(9)	-	1.424 (4)
S(1)—C(15)	-	1.754 (5)
S(2)—O(2)	-	1.602 (4)
S(2)—O(10)	-	1.420 (5)
S(2)—O(11)	-	1.426 (4)
S(2)—C(8)	-	1.752 (6)
O(1)—C(1)	1.202 (3)	1.199 (8)
O(2)—C(2)	1.403 (3)	1.408 (7)
O(3)—C(3)	1.427 (3)	1.414 (7)
O(4)—C(1)	1.339 (3)	1.338 (7)
O(4)—C(4)	1.478 (3)	1.465 (8)
O(5)—C(5)	1.425 (3)	1.390 (7)
O(6)—C(6)	1.425 (3)	1.413 (8)
O(7)—C(7)	1.424 (4)	1.482 (8)
C(1)—C(2)	1.509 (4)	1.515 (9)
C(2)—C(3)	1.533 (3)	1.515 (9)
C(3)—C(4)	1.519 (4)	1.508 (9)
C(4)—C(5)	1.522 (3)	1.524 (9)
C(5)—C(6)	1.526 (4)	1.518 (9)
C(6)—C(7)	1.511 (4)	1.502 (9)
O(7)—S(1)—O(8)	-	104.6 (2)
O(7)—S(1)—O(9)	-	110.1 (2)
O(7)—S(1)—C(15)	-	104.5 (2)
O(8)—S(1)—O(9)	-	117.9 (3)
O(8)—S(1)—C(15)	-	109.2 (3)
O(9)—S(1)—C(15)	-	109.6 (3)
O(2)—S(2)—O(10)	-	108.7 (2)
O(2)—S(2)—O(11)	-	102.6 (2)
O(2)—S(2)—C(8)	-	103.6 (2)
O(10)—S(2)—O(11)	-	121.5 (3)
O(10)—S(2)—C(8)	-	108.6 (3)
O(11)—S(2)—C(8)	-	110.2 (3)
S(2)—O(2)—C(2)	-	116.5 (3)
S(1)—O(7)—C(7)	-	118.0 (3)
C(1)—O(4)—C(4)	110.2 (2)	109.6 (5)
O(1)—C(1)—O(4)	122.5 (2)	123.4 (6)
O(1)—C(1)—C(2)	127.6 (2)	128.1 (6)
O(4)—C(1)—C(2)	109.9 (2)	108.5 (5)
O(2)—C(2)—C(1)	109.4 (2)	110.2 (5)
O(2)—C(2)—C(3)	115.7 (2)	117.3 (5)
C(1)—C(2)—C(3)	103.4 (2)	101.1 (5)
O(3)—C(3)—C(2)	111.5 (2)	109.9 (5)
O(3)—C(3)—C(4)	109.6 (2)	108.2 (5)
C(2)—C(3)—C(4)	101.4 (2)	100.1 (5)
O(4)—C(4)—C(3)	105.0 (2)	104.6 (5)
O(4)—C(4)—C(5)	105.2 (2)	108.1 (5)
C(3)—C(4)—C(5)	117.9 (2)	116.8 (5)
O(5)—C(5)—C(4)	108.9 (2)	113.3 (5)
O(5)—C(5)—C(6)	108.7 (2)	107.6 (5)
C(4)—C(5)—C(6)	114.4 (2)	113.1 (5)
O(6)—C(6)—C(5)	106.0 (2)	111.9 (5)
O(6)—C(6)—C(7)	109.0 (2)	112.2 (5)
C(5)—C(6)—C(7)	113.2 (2)	112.7 (5)
O(7)—C(7)—C(6)	111.3 (3)	108.5 (5)
S(2)—C(8)—C(9)	-	120.7 (5)
S(2)—C(8)—C(13)	-	119.7 (5)
S(1)—C(15)—C(16)	-	119.9 (4)
S(1)—C(15)—C(20)	-	119.0 (5)
O(1)—C(1)—O(4)—C(4)	179.2 (2)	170.0 (6)
O(1)—C(1)—C(2)—O(2)	−36.0 (4)	−22.8 (9)
O(4)—C(1)—C(2)—O(2)	143.5 (2)	154.9 (5)
O(2)—C(2)—C(3)—O(3)	−32.7 (3)	−44.7 (7)

O(3)—C(3)—C(4)—O(4)	−88.0 (2)	−79.8 (5)
C(5)—C(4)—O(4)—C(1)	−144.5 (2)	−143.0 (5)
O(4)—C(4)—C(5)—O(5)	−63.1 (3)	47.3 (7)
O(4)—C(4)—C(5)—C(6)	175.1 (2)	−75.5 (6)
C(3)—C(4)—C(5)—O(5)	−179.7 (2)	−70.3 (7)
C(3)—C(4)—C(5)—C(6)	58.5 (3)	166.9 (5)
O(5)—C(5)—C(6)—O(6)	78.2 (2)	−178.3 (5)
O(5)—C(5)—C(6)—C(7)	−41.2 (3)	54.1 (7)
C(4)—C(5)—C(6)—O(6)	−159.8 (2)	−52.4 (7)
C(4)—C(5)—C(6)—C(7)	80.8 (3)	179.9 (4)
O(6)—C(6)—C(7)—O(7)	54.1 (3)	−69.0 (7)
C(5)—C(6)—C(7)—O(7)	171.7 (2)	58.4 (6)

Table 3. *Hydrogen-bonding geometry* (Å, °) *for compounds* (I) *and* (II)

D—H···*A*	H···*A*	*D*···*A*	*D*—H···*A*
(I)			
O(2)—H(O2)···O(6^{i})	2.635 (3)	1.83 (4)	174 (4)
O(3)—H(O3)···O(2^{ii})	2.755 (3)	1.99 (3)	161 (3)
O(5)—H(O5)···O(7^{iii})	2.683 (3)	1.91 (4)	158 (4)
O(6)—H(O6)···O(3^{iv})	2.710 (3)	1.92 (3)	175 (3)
O(7)—H(O7)···O(1^{v})	2.913 (3)	2.20 (3)	168 (4)
(II)			
O(3)—H(O3)···O(1^{iii})	2.750 (6)	2.09 (6)	158 (6)
O(5)—H(O5)···O(3)	2.718 (6)	2.17 (7)	124 (6)

Symmetry codes: (i) $x, 1+y, z$; (ii) $\frac{1}{2}+x, \frac{3}{2}-y, 1-z$; (iii) $x-1, y, z$; (iv) $\frac{1}{2}+x, \frac{1}{2}-y, z$; (v) $x+1, y-1, z$.

Except for the two CH_3 groups in (II), the H atoms were located in difference Fourier maps and refined isotropically. Note that C(21) of one of the CH_3 groups has a large temperature parameter. There is a significant difference between the *R* values for the two possible enantiomorphic structures of (II). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELX76* (Sheldrick, 1976). Molecular geometry calculations: *PLATON* (Spek, 1990). Molecular graphics: *PLUTO* (Motherwell & Clegg, 1978).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71663 (31 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1093]

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2,6-Dimesyl-D-*manno*-hexono- (III), 2,6-Dimesyl-D-*allo*-hexono- (IV) and 2,6-Dimesyl-D-*gulo*-hexono-1,4-lactone (V)

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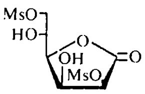
Abstract

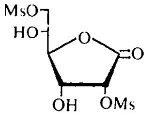
The geometries of the lactone rings in the three structures are similar. Differences between $C_8H_{14}O_{10}S_2$ (III), $C_8H_{14}O_{10}S_2$ (IV) and $C_8H_{14}O_{10}S_2$ (V) arise in the conformation of the side chain and in the crystal packing of the structures.

Comment

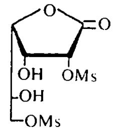
The present structure analyses are part of an investigation of tosylated and mesylated 1,4-lactones (Søtofte, 1994). The di-*O*-mesylation of D-*manno*-, D-*allo*- and D-*gulo*-1,4-lactones gave the 2,6-di-*O*-mesylates (III), (IV) and (V) in 42, 16 and 27% yields, respectively. The yields of the di-*O*-mesylates are lower than those of the di-*O*-tosylates, indicating lower selectivity. All three compounds were prepared by literature methods (Lundt & Madsen, 1992).



(III)



(IV)



(V)

Compound (III) was recrystallized at room temperature from acetonitrile, while compounds (IV) and (V) were recrystallized from ethyl acetate. Unfortunately, the structure determined for (III) is rather poor, as no larger and more suitable crystals could be obtained by recrystallization. The bond lengths and angles that are listed in Table 2 agree well with those observed in related structures. The labelling of the atoms is shown in Fig. 1. The lactone rings are nonplanar.